

(PATENT)

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of:
Roland Deckwer et al.

Application No.: 10/734,828

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Art Unit: 1616

For: OIL SUSPENSION CONCENTRATE

Examiner: A. N. Pryor

DECLARATION

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

I, Dr. Roland Deckwer, state that I reside at Königsteinerstrasse 92a, D-65929 Frankfurt/Main; I am a citizen of the Federal Republic of Germany; that I am familiar with the subject matter and the prosecution of the instant application Serial No. 10/734,828 filed December 12, 2003, entitled "Oil Suspension Concentrate"; that I consider myself qualified by my education, knowledge and experience in physical chemistry and formulation technology to make this Declaration; and that I have made the following observations:

1. The instantly claimed invention is directed to novel oil suspension concentrates formulations comprising a) one or more herbicidially active compounds from the group of the sulfonamides, b) one or more safeners, c) one or more organic solvents, and d) one or more sulfosuccinates.

2. The task was to deliver oil suspension concentrates with improved chemical and physical stability in manufacture, storage, handling and application.

3. The following tests were carried out under my supervision and direction. The tests were conducted in the same manner as described in the specification. The names and abbreviations used in the examples have the following meanings:

Iodosulfuron	=	methyl 4-iodo-2-[3-(4-methoxy-6-methyl-1,3,5-triazin-2-yl)ureidosulfonyl]benzoate sodium
Mesosulfuron	=	2-[(4,6-dimethoxypyrimidin-2-ylcarbamoyl)sulfamoyl]- α -(methanesulfonamido)-p-toluic acid sodium
Foramsulfuron	=	1-(4,6-dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)-5-formamidophenylsulfonyl]urea sodium
Foramsulfuron (acid)	=	1-(4,6-dimethoxypyrimidin-2-yl)-3-[2-(dimethylcarbamoyl)-5-formamidophenylsulfonyl]urea
Ethoxysulfuron	=	1-(4,6-dimethoxypyrimidin-2-yl)-3-(2-ethoxyphenoxysulfonyl)urea sodium
Amidosulfuron	=	1-(4,6-dimethoxypyrimidin-2-yl)-3-mesyl(methyl)sulfamoylurea sodium
Propoxycarbozon	=	methyl 2-[(4,5-dihydro-4-methyl-5-oxo-3-propoxy-1H-1,2,4-triazole-1-carboxamido)sulfonyl]benzoate sodium
Flucarbazon	=	4,5-dihydro-3-methoxy-4-methyl-5-oxo-N-[2-(trifluoromethoxy)phenylsulfonyl]-1H-1,2,4-triazole-1-carboxamide sodium
A21.1	=	4-[(4,5-dihydro-3-methoxy-4-methyl-5-oxo-1H-1,2,4-triazol-1-yl)carbonylsulfamoyl]-5-methylthiophene-3-carboxylic acid
Thiensiulfuron	=	methyl 3-(4-methoxy-6-methyl-1,3,5-triazin-2-ylcarbamoylsulfamoyl)thiophen-2-carboxylate sodium
Mefenpyr	=	diethyl (RS)-1-(2,4-dichlorophenyl)-5-methyl-2-pyrazoline-3,5-dicarboxylate
Isoxadifen	=	4,5-dihydro-5,5-diphenyl-1,2-oxazole-3-carboxylic acid
Cyprosulfonamid	=	N-[4-(cyclopropylcarbamoyl)phenylsulfonyl]-2-methoxybenzamide
Emulsogen® EL-400	=	ethoxylated castor oil, Clariant
Emulsogen® EL-360	=	ethoxylated castor oil, Clariant
Triton® GR7-M E	=	dioctyl sulfosuccinate sodium, Dow
Genapol® V4739	=	ethoxylated tridecyl alcohol, Clariant
Genapol® XM150	=	ethoxylated tridecyl alcohol, Clariant
Genapol® X060	=	ethoxylated tridecyl alcohol, Clariant
Genapol® PF10	=	tri-block copolymer of ethylene oxide and propylene oxide, Clariant
Emcol® P1860	=	dodecyl benzene sulfonate calcium salt, Akzo Nobel
Solvesso® 200	=	aromatic mineral oil (boiling point 219-281°C), Exxon
Edenor® MESU	=	rape seed oil methyl ester, Cognis
Bayol® 85	=	aliphatic mineral oil (boiling point 330-440°C, Exxon
Jeffsol® PC	=	propylene carbonate, Huntsman
Bentone® 34	=	sheet silicate organic modified, Elementis
Bentone® 38	=	sheet silicate organic modified, Elementis
Bentone® SD-1	=	sheet silicate organic modified, Elementis

Thixatrol® ST	=	polymeric modified castor oil, Elementis
before bead milling	=	sample analyzed by HPLC (High Performance Liquid Chromatography) direct after mixing of all components
after bead milling	=	sample analyzed by HPLC direct after grinding of the mixed components, e.g. by bead milling using 1 mm glass beads
after storage (8w40°C)=	=	sample analyzed after a storage period of eight weeks at 40°C by HPLC
d50 / d90	=	particle / crystal diameter in µm by light scattering (LS Particle Size Analyzer, Coulter) - d50 or d90 indicates the volume weighted percentage of the particles with a diameter below the noted diameter
g/L	=	gram per liter
%w/w	=	percentage weight by weight

4. Example 1 relates to the manufacturing process of oil suspension concentrates. Example 1 shows the effect of Triton®GR-7M E (sulfosuccinate) on the chemical stability of Iodosulfuron (sulfonamide) while grinding.

Example 1 - Table 1

Chemical stability of Iodosulfuron while manufacturing of the oil suspension concentrate (bead milling)

	Example 1.1	Example 1.2
	Concentration in g/L	
Iodosulfuron	5.00	5.00
Mefenpyr	15.00	15.00
Triton®GR-7M E	--	25.00
Edenor® MESU	62.64	37.38
Genapol® PF10	5.00	5.00
Emulsogen® EL-400	5.00	5.00
Thixatrol® ST	1.00	1.00
concentration of Iodosulfuron (measured by HPLC)		
before bead milling	5.00	5.00
after bead milling	3.50	4.89

5. Example 2 relates to the chemical stability while manufacturing and storage of the oil suspension concentrate.

Example 2 - Table 2

Variation of active ingredient loading and chemical stability while manufacturing and after storage (8w40°C)

	Exp. 2.1 in %w/w	Exp. 2.2 in %w/w	Exp. 2.3 in %w/w
Iodosulfuron	5.00	10.00	12.00
Mefenpyr	15.00	30.00	36.00
Solvesso® 200	42.00	28.00	20.00
Jeffsol® PC	1.00	0.50	--
Triton® GR-7M E	25.00	20.00	20.00
Genapol® V4739	5.00	5.00	5.00
Genapol® PF10	3.00	3.00	3.00
Emulsogen® EL-400	3.00	3.00	3.00
Bentone® 34	1.00	0.50	--
concentration of Iodosulfuron (measured by HPLC)			
before bead milling	5.00	10.00	12.0
after bead milling	4.97	9.92	11.5
after storage (8w40°C)	4.73	9.73	11.5

6. Example 3/Example 4 summarizes different variations of oil suspension concentrates:

Sulfonamid -	Foramsulfuron, Mesosulfuron, Ethoxysulfuron, Amidosulfuron, Propoxycarbazon, Flucarbazon, A21.1, Thifensulfuron;
Safener -	Mefenpyr, Isoxadifen, Cyprosulfonamid;
Organic solvent -	Solvesso® 200, Edenor® MESU, Bayol® 85, Jeffsol® PC;
Dispersant/Emulsifier -	Genapol® PF10, Emulsogen® EL-400, Emulsogen® EL-360, Genapol® 4739, Genapol® XM150; Genapol® X060, Emcol® P1860;
Rheological additive -	Thixatrol® ST, Bentone® SD-1, Bentone® 38.

All oil suspension concentrates summarized in Table 3, show good chemical and physical stability while storage (8w40°), and confirm that the stability is given for the claimed oil suspension concentrates.

Example 3/ Example 4 – Table 3
Variation of components

Example	3.2 in %w/w	4.1 in %w/w	4.2 in %w/w	4.3 in %w/w	4.4 in %w/w	4.5 in %w/w	4.6 in %w/w	4.7 in %w/w	4.8 in %w/w	4.9 in %w/w
Foramsulfuron (acid)		3.00								
Mesosulfuron			4.50							
Ethoxysulfuron				2.50						
Amidosulfuron					10.00					
Propoxycarbazon						3.00				
Flucarbazon							3.00			
A21.1								1.00	1.00	1.00
Thifensulfuron	8.00									
Mefenpyr	24.00		12.00		20.00	9.00	10.00	5.00		
Isoxadifen		3.00		9.00					2.00	
Cyprosulfonamid										0.50
Triton®GR-7M E	25.00	10.00	10.00	12.50	20.00	15.00	15.00	25.00	20.00	20.00
Solvesso® 200		66.50	46.30	58.00	37.50		48.40	50.00	58.00	64.20
Edenor® MESU	32.00		10.00			10.00	10.00			
Bayol® 85						46.60				
Jeffsol® PC						0.20	0.50	1.00	1.00	1.00
Genapol® PF10	5.00				2.50			2.00	2.50	
Emulsogen® EL-400	5.00	3.00						4.00		
Emulsogen® EL-360			3.00	3.00	2.00				3.50	3.00
Genapol® V4739		10.00		10.00		15.00		8.00	8.00	
Genapol® XM150			10.00		8.00		12.00			
Genapol® X060										
Emcol® P1860		2.00	2.00	2.50				2.00	2.00	8.00
Thixatrol® ST	1.00									
Bentone® SD-1			2.20					2.00		
Bentone® 38		2.50		2.50					2.00	2.30
Bentone® 34						1.20	1.10			

7. Example 5 shows the influence of Triton GR-7M E (sulfosuccinate) on the crystal size of the oil suspension concentrate accessible by intensive bead milling. A low crystal size is necessary to make suspension concentrates stable against sedimentation. Sedimentation makes the liquid handling difficult and could lead to wrong dosages. Beside this, the increase in crystal size could lead to nozzle blocking while application and increases cleaning effort of application equipment.

Example 5 – Table 4
Crystal size accessible by intensive bead milling

	Example 5.1 in %w/w	Example 5.2 in %w/w
Foramsulfuron	2.32	2.32
Isoxadifen	2.32	2.32
Atplus 309 F	9.00	9.00
Emcol P 1860	4.00	4.00
Emulsogen EL-400	4.00	4.00
Solvesso 200	50.86	75.86
Bentone 38	2.50	2.50
Triton GR-7M E	25.00	--
crystal size:		
d50	1.21 μm	22.6 μm
d90	2.41 μm	62.7 μm

8. Example 6 shows the influence of impurities of water on the crystal size of the oil suspension concentrate accessible by intensive bead milling. Oil suspension concentrates have to be robust in a certain range of water content, because the water content of the raw materials will differ from batch to batch.

Example 6 – Table 5

Dependency of crystal size accessible by intensive bead milling on the water content

	Example 6.1 in %w/w		Example 6.2 in %w/w	
Foramsulfuron (acid)	2.32		2.32	
Isoxadifen	2.32		2.32	
Atplus 309 F	9.00		9.00	
Emcol P 1860	4.00		4.00	
Emulsogen EL-400	4.00		4.00	
Solvesso 200	7.00		32.00	
Edenor MESU	43.75		43.75	
Bentone 38	2.50		2.50	
Triton GR-7M E	25.00		--	
addition of water	none	+ 1.0 %w/w	none	+ 1.0 %w/w
crystal size:				
d50	1.39 μm	6.05 μm	1.52 μm	1.36 μm
d90	3.39 μm	64.0 μm	5.03 μm	3.49 μm


9. Example 7 shows the influence of impurities of water on the physical stability of the oil suspension concentrate. Oil suspension concentrates have to be robust in a certain range of water content, because the water content can increase while storage and handling.

Example 7 – Table 6

Dependency of the physical stability on the water content after storage

	addition of water	appearance / viscosity after storage (8w40°C)
Example 6.2 (without Triton GR-7M E/ sulfosuccinate)	none	thin easily flowable
	+1.0 %w/w	thick, lumpy hardly flowable
Example 6.1 (with Triton GR-7M E/ sulfosuccinate)	none	thin easily flowable
	+1.0 %w/w	thin easily flowable

I declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing there from.

Signed: 

Name: Dr. Roland Deckwer

Date: 20.06.2008